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NO DRAWINGS.

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COMPLETE SPECIFICATION.

Hydrazine.

We, FISONS INDUSTRIAL CHEMICALS LIMITED, a British Company, of Willows Works, Derby Road, Loughborough, Leicestershire, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a process for obtaining hydrazine from methyl ethyl ketazine.

In the Raschig synthesis of hydrazine, hypochlorite is reacted with ammonia to form chloramine which is reacted with further ammonia to form hydrazine. It has been proposed to increase the yields obtained by this process by performing the process in the presence of a carbonyl compound which gives a hydrazone or azine from which hydrazine can be recovered. If the carbonyl compound is methyl ethyl ketone an azine is formed which is substantially immiscible with water, but in spite of this it has been found that methyl ethyl ketone azine can surprisingly be readily converted into hydrazine by suitable treatment with water. One such conversion is described and claimed in our copending Application No. 9210/65 (Specification No. 1,129,613).

The present invention provides a process for the preparation of hydrazine which comprises feeding to an intermediate point of a fractionating column operated at 3 to 15 atmospheres above atmospheric pressure and at a temperature between 150°C. and 200°C., a two phase mixture containing 1.5 to 2.5 parts by weight of water and 1 part by weight of methyl ethyl ketazine, whereby a mixture containing methyl ethyl ketazine, methyl ethyl ketone hydrazone, hydrazine and methyl ethyl ketone is formed, recovering methyl ethyl ketone by rectification of the hydrazine-containing mixture in a zone of the fractionating column above the feed point, stripping the azine and the hydrazone from the hydrazine-containing mixture, in a zone of the fractionating column at a distance below the feed point equivalent to 6 to 8 theoretical plates, the stripping being effected by steam, and recovering an aqueous solution containing essentially hydrazine from the sump, the residence time in the sump being such that residual amounts of ketazine and hydrazone are converted to hydrazine therein.

Preferably the two phase mixture is heated to a temperature in the range 150°C. to 200°C. before it is fed to the fractionating column.

Preferably the residence time in the sump is 1 to 2 hours.

The aqueous solution containing hydrazine is preferably distilled at atmospheric pressure to separate the minor contaminants such as hydrazone.

The process of the present invention is illustrated by the following example.

Example

The apparatus used consisted of a 5 litre still fitted with two 3 foot sections of 2 inch internal diameter columns packed with stainless steel knitmesh. A magnetically controlled reflux ratio divider was positioned on top of the packing and below the condenser section.

2.5 Litres of 10% w/w hydrazine were heated in the still to 180°C. Methyl ethyl ketone (1100 millilitres per hour) and water (2160 millilitres per hour) were mixed in a T piece, preheated to 160°C and pumped to the middle of the packed column. The sump temperature was maintained at 180°C. and the pressure at 145 pounds per square inch

[Price [illegible])]

gauge. The column was operated at a boil-up rate of 6.4 litres per hour. The product was continuously removed from the still at 1885 millilitres per hour, giving a sump residence time of 1.35 hours. The liberated methyl ethyl ketone was removed as its azeotrope at a reflux ratio of 4:1. This distillate contained 1.5% of the azine input and was recycled to the azine synthesis section.

The product was obtained in 98% yield as a 10% w/w solution of hydrazine containing 0.23% of methyl ethyl ketone hydrazone.

The hydrazine was separated from the trace of hydrazone by simple distillation at atmospheric pressure.

WHAT WE CLAIM IS:—

1) A process for the preparation of hydrazine which comprises feeding to an intermediate point of a fractionating column operated at 3 to 15 atmospheres above atmospheric pressure and at a temperature between 150°C. and 200°C., a two phase mixture containing 1.5 to 2.5 parts by weight of water and 1 part by weight of methyl ethyl ketazine, whereby a mixture containing methyl ethyl ketazine, methyl ethyl ketone hydrazone, hydrazine and methyl ethyl ketone is formed, recovering methyl ethyl ketone by rectification of the hydrazine-containing mixture in a zone of the fractionating column above the feed point, stripping the azine and the hydrazone from the hydrazine-containing mixture, in a zone of the fractionating column at a distance below the feed point equivalent to 6 to 8 theoretical plates, the stripping being effected by steam, and recovering an aqueous solution containing essentially hydrazine from the sump, the residence time in the sump being such that residual amounts of ketazine and hydrazone are converted to hydrazine therein.

2) A process as claimed in claim 1 wherein the two phase mixture is heated to a temperature in the range 150°C. to 200°C. before it is fed to the fractionating column.

3) A process as claimed in claim 1 or claim 2 wherein the residence time in the sump is 1 to 2 hours.

4) A process as claimed in any of the preceding claims wherein the aqueous solution containing hydrazine recovered from the sump is distilled at atmospheric pressure.

5) A process for the preparation of hydrazine as claimed in claim 1 substantially as hereinbefore described.

6) Hydrazine when prepared by the process as claimed in any of the preceding claims.

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